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**MICRO-SPECTROSCOPIC OBSERVATIONS.**

BY F. J. KEELEY.

Since Sorby devised the micro-spectroscope, nearly fifty years ago, an immense amount of work has been done with this instrument, but principally in the study of organic coloring matters in solution or section, which could be examined by transmitted light. It was understood that the micro-spectroscope could likewise be employed in examining objects by reflected light, but this phase of its utility has been almost completely neglected. With transmitted light, it produces results from very minute quantities of material, but otherwise offers little advantage over an ordinary spectroscope of low dispersion. With reflected light, it is also applicable to the study of small quantities, but has a number of additional advantages. Absorption bands are usually more distinct; a small crystal on a mineral specimen or a gem embedded in an opaque setting may be examined without disturbing or damaging it; and phenomena such as iridescence on opaque substances may be studied.

The microscope employed in connection with this instrument should always be a binocular, which permits the object to be examined through one tube while the spectroscopic ocular is applied to the other. The illumination should preferably be rather brighter and whiter than is generally necessary for the examination of opaque objects, and is best concentrated by means of a parabolic silvered reflector attached to the objective. For preliminary examinations, the older form of micro-spectroscope, as made by Browning or Beck, is preferable, as very faint bands are more readily noted in its short bright spectrum, but for further study and recording the spectra, the Zeiss model, with photographed scale, is more desirable.

There are two classes of spectra that can be advantageously studied under the conditions specified, those showing absorption bands, which permit the identification of certain minerals and gems; and interference spectra, which assist in determining the cause of luster and iridescence.

Of the first class, the characteristic spectrum of didymium is the best known and most useful. While didymium has been resolved into two elements, neodymium and praseodymium, for the present

purpose the absorption bands which have become classical as those of didymium can be regarded as indicating the presence of the whole group, including cerium, lanthanum, erbium and terbium, the last two of which likewise have characteristic spectra of their own, not usually visible in minerals to be considered.

Under most favorable conditions, the spectrum of "didymium" contains nine absorption bands, centering at about the following wave lengths, .675, .622, .591, .575, .532, .521, .508, .480, .471 micron, the first two of which, in the red, are not usually visible in a solution or section examined by transmitted light, but quite distinct when a crystallized salt or a mineral such as lanthanite is viewed by reflected light. Position of bands is given as determined by the photographed wave-length scale on Zeiss micro-spectroscope, which was itself adjusted on the D line from a sodium flame, and is probably sufficiently accurate for the purpose. For reference, all spectra should be recorded diagrammatically on paper ruled with lines corresponding to the scale, showing width of band, whether edges are sharp or misty and whether the darkest part of band is central or eccentric.

In determining the presence of this element, it is of course unnecessary that all the bands be visible. If two or three, including the strongest at junction of yellow and green, can be seen and their position accurately measured, it is sufficient.

Many of the minerals characterized by the "didymium" spectrum occur in microscopic crystals, sometimes not sufficiently well developed to determine with certainty, but if the presence of these absorption bands demonstrates that the mineral is one of the comparatively few which contain the cerium group, there will always be sufficient indications of its identity from color, luster or such few crystal faces as may be present. In this manner, I have been able to confirm the discovery of monazite in Delaware County, where but a few small crystals have been found, and likewise to identify microscopic crystals of parisite from a new locality in New England.

Another element which shows a characteristic spectrum in most of its compounds is uranium, the several equidistant absorption bands in the blue end of spectrum, assisting in determining poorly crystallized microscopic occurrences of such minerals as autunite and torbernite.

A mineral that has afforded material for extensive study of spectra is zircon, which has long been known to show absorption bands, the cause of which seems unexplained. These bands center at about wave lengths .687, .650, .615, .586, .561, .538, .508, .478 and .455

micron, and have been attributed to the presence of the cerium group of elements, but a comparison of the spectra will demonstrate that this cannot be the case, as not a single band occupies a similar position. Another writer refers to them as "the characteristic spectrum of zircon," which is likewise incorrect, as a large proportion of the varieties of zircon show no trace of them. Neither are they due to the coloring matter which gives various tints to this mineral, for except in the case of the red variety, known as hyacinth, these colors are discharged by heating without affecting the spectrum. Having examined more than fifty varieties and colors of zircon, natural crystals and cut gems, many of them both before and after bleaching by heat, can generalize somewhat as follows: In no case have I seen a trace of absorption bands in the spectra of the red hyacinth variety or in the colorless microscopic crystals which can be picked out of magnetic and gold-bearing sands from many localities. They are generally likewise absent in crystals of brown and gray tints, while in the transparent gem varieties from India and Ceylon, those of the characteristic pinkish-brown and yellow tints sometimes show a complete spectrum, sometimes only a few bands and occasionally none whatever. When bleached by heating, the spectrum is unchanged. The strongest and most distinct absorption bands are shown by those stones more or less green in color which become very pale blue after heating. A similar strong spectrum is shown by the microscopic bluish zircons occurring in Vesuvian lava, but no trace of it in the opaque white varieties from the Laacher See and the Azores. It would therefore seem that the spectrum sometimes shown by this mineral is due to the same cause, whatever it may be, that produces a faint blue tint, whether this is originally visible or veiled by other coloring matter which can be bleached by heat. As at least a few of the bands are almost always visible in the spectra of the varieties of zircon employed as gems, except the red, they afford a means of identifying the stone when set. If loose, its high specific gravity is sufficient for the purpose.

Red garnets likewise have a readily distinguished absorption spectrum, including three broad and one narrow band, centering about wave length .618, .578, .520 and .500 micron. This is unquestionably due to the color, possibly manganese, as it is not shown by garnets of other tints than red, but is remarkably constant in all the varieties used as gems. Examination of over a hundred from all parts of the world, including spessartite, almandite and pyrope, as well as varieties, such as the beautiful "rhodolite" from North Carolina, which include both the almandite and pyrope molecules, showed no important ex-

ception, hence this spectrum will serve to identify a red garnet with practical certainty. Red spinels show no absorption bands in their spectra.

The ruby exhibits an interesting spectrum consisting of a very narrow bright line between two absorption bands, at wave length .69 micron in the extreme red. A careful search of the literature of the subject has enabled me to find no evidence that this spectrum had been previously observed and recorded, but whether new or old, it is most characteristic, not only of typical rubies, but of every variety of corundum that contains a trace of red in its coloration, including the various shades of pink and amethystine sapphires, even when so pale as to appear colorless with artificial light, star rubies, Montana sapphires of pinkish cast and even corundum from North Carolina and other localities, which has a pink tint. The remainder of the spectrum may vary, all except the red being absorbed by deep colored oriental rubies, or all the colors visible in light tinted varieties, but the narrow bright band in the red remains the same.

This spectrum is unquestionably due to chromium, the coloring matter of the ruby, as it is likewise found in examining artificial rubies which owe their color to the same element. It affords an infallible test for distinguishing a ruby from a garnet, spinel or tourmaline of similar color, and is applicable to stones in deep mountings or even if covered by a plate of glass. The artificial ruby is the only other stone giving a similar spectrum, and it can readily be distinguished by means of the microscope alone.

A number of other precious stones exhibit fainter but nevertheless characteristic spectra. Blue and violet spinels show two narrow and fairly sharp lines in the yellow and green, and, as in the case of the ruby, this spectrum is very persistent, the bands being scarcely less visible when the stone is almost colorless than from one of deep color. It is particularly available as a means for easily distinguishing pale amethystine spinels from sapphires of identical color frequently found mixed in same paper of Indian cut stones.

Among green gems, the emerald exhibits a faint line in the red and a broader shadowy band occluding the orange and yellow. Green garnet or demantoid, often sold as "olivine," has two very faint bands in the red, which coalesce into one when the stone is deep in color, while the true olivine or peridot shows two bands in the blue and violet, which, although rather broad and well-defined, are usually extremely difficult to see, owing to the comparatively small amount of light of the shorter wave lengths transmitted by this mineral.

In studying the luster and iridescence shown by various natural objects, it must first be considered to what causes they may be due, which include:

Simple reflection, as in the case of minute pyrite crystals sometimes used in jewelry.

Refraction and dispersion, generally accompanied by internal reflection and sometimes by absorption, which account for the brilliancy and colors shown by dew drops, frost crystals and most cut gems.

Scattering of light from microscopic particles, to which the blue color of the sky is due.

Polarization, which frequently accompanies the other phenomena, but is rarely responsible for natural colors. If translucent tourmaline enclosed in quartz or mica happens to be examined in the polarized light from the sky at right angles to the direction of the sun's rays, it may act as an analyzer, and if the film of the including mineral between it and the source of light be of suitable thickness, show bright colors.

Diffraction, or the interference resulting from fine, uniformly spaced lines or dots, which has been credited with being the cause of many iridescent effects, with which it rarely has anything to do. The small crustacean *Sapphirina*, which is said to sparkle like a gem when swimming in the sun light, has a shell covered with fine markings similar to those on *Pleurosigma angulatum*, which no doubt cause these brilliant colored reflections by diffraction, and the chatoyance of star sapphires and cat's-eyes may be due largely to diffraction resulting from the symmetrically arranged inclusions. It must not be overlooked, however, that to produce color effects by diffraction, the light must come from but one direction, and the color will vary through the entire spectrum with changes in the angle of incidence.

There is one more cause to which these effects may be attributed, and to which investigation will show that practically all iridescence is due, and that is the interference produced by reflection from thin films, which can be advantageously studied with the micro-spectroscope. Such interference colors generally show dark bands in the spectrum, one in the lower order colors produced by thin films and two or more as the films become thicker so that additional wave lengths interfere. For comparison, records should be made of the spectra of all the brighter colors, which can be done by observing them in the "Newton's rings" produced between two surfaces of glass or by blowing a bubble of melted glass until it bursts when the thin edges will answer the same purpose.

If the natural iridescent surfaces shown by many minerals be now examined, similar interference bands will be noted in the spectra, assuring us of the cause of iridescence and permitting the thickness of the film to be calculated if the refractive index of the mineral be known. This is shown particularly well on the iridescent surfaces which sometimes appear on dendritic inclusions of magnetite in mica from Delaware Co. Penna. One such surface of uniform pink color gave a spectrum with two dark bands, centering at about wave lengths .545 and .457 micron, corresponding to a red of the fifth order, and indicating a thickness, if we assume the film to be a hydrate of the composition of goethite, of about .53 micron or .000021 inch. In a similar manner the colors reflected by scales of hematite, to which the chatoyance of varieties of feldspar known as "sun stone" is due, can be proved to result from the thinness of the films. The oligoclas from Tvedestrand, Norway, frequently contains films of sufficient size and uniformity of thickness to produce sharp interference bands. Some hypersthene as well as other minerals showing aventurine reflections contain similar inclusions, but not always of the same mineral. The common aventurine or gold stone used in jewelry is an artificial glass which owes its brilliancy to reflections from enclosed crystals of metallic copper.

There is no class of objects furnishing better examples of brilliant iridescence than the scales of butterflies, and this is generally attributed to the fine lines and markings with which they are covered, which cannot be the case, as such markings could only cause color effects by diffraction, and the limitations to this cause have already been mentioned. It is true that diffraction effects may be obtained from these scales when held in a certain position with respect to the light, particularly when they have been mounted on glass for examination by transmitted light, but the colors thus produced which undergo changes according to the angle of the illumination are not the characteristic colors of the scales as seen in position on the insect, which are equally well shown in diffused light. Furthermore, it will be found that in many butterflies the scales showing metallic colored reflections are not the ordinary lined scales, but are apparently specialized for their purpose, showing no fine markings, but merely rather coarse longitudinal corrugations. When examined under the micro-spectroscope, most butterfly scales are not sufficiently uniform in color to give pronounced interference bands, but a darkening of the spectrum in some one position may usually be noted, and in the particularly brilliant blue spots on the wings of *Papilio paris*, a distinct black band is shown, having its center

at about wave length .588 micron. The position and width of this band correspond to that in the spectrum of blue of the third order, and accordingly may be accepted as indicating a film about .58 micron in thickness, the film being assumed to be of air or gas enclosed within the chitine of the scale, and not consisting of chitine itself, in which case it would be over a third thinner.

The reason for this assumption lies rather in analogy with the results obtained from study of the scales of beetles than in the behavior of the butterfly scales themselves. It is well known that if a portion of a diamond beetle is mounted in balsam, the iridescent reflections from the scales are more brilliant than when examined dry, but if the scales be scraped from such a beetle and then mounted in balsam, most of them will become perfectly colorless, while here and there may be one still retaining its iridescence. A further examination will show that such colored scales are perfect, while the others have all been more or less broken in removing them, indicating that the iridescent film occupied an internal interval, which was penetrated by the balsam in all broken scales, but remained unimpaired as long as no such penetration could be effected.

If the iridescent feathers of birds be next examined, the resemblance of their brilliant metallic reflections to those of butterfly scales would seem to justify the expectation that this is due to a similar cause, but this predication will be contradicted by an examination of their spectra. For instance, the spectrum from a ruby-tinted, iridescent humming-bird feather shows no dark bands, but on the contrary a single bright band, including part of the red and orange, and reference to the record of spectra previously made proves that there can be no color resulting from interference caused by a single film that could produce such a spectrum. We can, however, readily understand the probable cause of this color by first considering to what the colors in precious opal are due, a subject that has been elucidated by high authorities.

In selecting opals for this investigation, those presenting patches of uniform color are to be preferred, and the best will be found among the so-called black opals which have recently become popular as gems. I have examined a large number of these, both in polished specimens and microscopic sections, as a result of which it is evident that they consist of precious opal, which by some convulsion of nature has been shattered to fragments, which were subsequently re-cemented by a further deposition of opal of a gray or black color, which serves to render the reflections more brilliant by cutting out all extraneous



light. In the absence of such black opals, a good substitute can be prepared by grinding a thin section of ordinary precious opal and backing it with India ink. Some opals in a dark matrix, likewise serve admirably.

On examining the colored reflections with a micro-spectroscope, instead of a spectrum with a dark band in the position of the complementary color, which characterizes the interference spectrum from a thin film, we find only a narrow band of same color as that shown visually. This has been explained by Lord Rayleigh as being due to the successive action of numerous parallel films of a thickness of the same order as the wave lengths of light, an explanation first applied to the similar colors reflected by certain crystals of potassium chlorate, in which the films are known to be due to repeated twinning. To what they are due in opal is not known, but under favorable conditions they may become quite visible, although I have hitherto been unable to distinguish them in sections at right angles to the laminæ. In one specimen of opal reflecting a brilliant green color have been able to count laminæ 38,000 to the inch, and as they appeared to be at an angle of about  $45^{\circ}$  to the plane of the section this would correspond to over 50,000 to the inch at right angles to the plane of lamination.

The manner in which the colors of opal have been accounted for justifies the application of same explanation to the iridescent feathers previously referred to, which show bright-band spectra, although I have seen none in which the band was as sharp and narrow as in the opal, whose colors are probably the purest shown by any natural object. It will not be safe, however, to apply the same reasoning to all feathers, for Nature does not hesitate to use various means to the same end. An illustration of this will be found in another humming-bird feather, which in general structure corresponds to that already mentioned, except that the reflections are blue in color, but at the end of each pinnule there is a single filament which glows with a most brilliant ruby color and shows a spectrum not unlike that of the other ruby-tinted feather, but on examining a balsam-mounted specimen with transmitted light, the color and spectrum are the same as by reflected light, proving that it is due to absorption and not to interference. The brilliancy will be accounted for by examining sections of the filaments, which prove to be somewhat rounded trigonal prisms, with an edge facing outward, so that light striking either side is returned by internal reflection from the back; or, in other words, Nature has here employed the same method that a skilful lapidary would

use in cutting a cabochon ruby to bring out its brilliant color to best advantage.

There are other minerals beside opal which show colored reflections due to successive laminae. The "moon stone" from Delaware Co., Penna., consists of triclinic feldspar, albite or oligoclase, and the opalescence is shown on the face parallel to the laminae due to repeated twinning, and only on such specimens in which the laminae are extremely fine. The reflections from this mineral vary from a pearly-white to sky-blue, but in the closely related labradorite they assume all tints, although never as pure as in the case of the opal, probably owing to the less uniform spacing of the laminae, although these are thin enough to account for the colors. Labradorite sometimes likewise shows entirely different reflections corresponding to those of "sun stone" and due to the same cause, interference resulting from single thin films of included minerals.

The chatoyance of Ceylon "moon stone," frequently cut as a gem, cannot be accounted for in a similar manner, as it consists of adularia, a variety of orthoclase, which does not contain twin laminations, but on examining a section at right angles to the opalescent face, with polarized light and comparatively high powers, a very fine micro-perthite structure, due to intergrowth with a small percentage of another feldspar, becomes apparent. The micro-spectroscope is of no help here, as the spectra show no characteristic appearances, but the fact that the reflected blue light from one of these "moon stones" is partially polarized raises the question whether it may not be due, at least in part, to the scattering of light such as causes the blue color of a clear sky, and the microscopic texture of the mineral seems better calculated to produce this effect than it does to produce the color by any known form of interference.

If authorities be consulted as to the cause of the luster of pearls, the explanation given will be generally found to be that it is due to the breaking up of the light by reflections from minute corrugations with which the surface of the pearl is covered. This explanation, originally given by Sir David Brewster, has been copied by all the writers in whose works I have been able to find a reference to the subject. Brewster made an extended investigation of the phenomena shown by pearl shell in the form of plates and sections, and finding that light from a contracted source produced undoubted diffraction effects when reflected from the surfaces of such pieces of pearl, concluded that the fine wavy or serrated parallel lines which he discovered were largely responsible for the pearly luster, without apparently

giving due consideration to the facts that this luster was likewise present in diffused light and that the lines described might not necessarily be present in an undamaged pearl, but were due to cutting a section through the laminae which produced them. Brewster's own description of his investigations proves that he did not overlook the light reflected from the laminae of the pearl, but he does not appear to have regarded it as differing from ordinary reflected light, and paid most attention to the effects of diffraction, which he distinguishes from the reflected light by the term "communicable colors" because they could be communicated by pressure to another surface of softer material. Later writers, however, seem to have regarded only the lines whose influence on the true luster of a pearl is negligible.

I have one shell of the so-called pearl oyster, *Meleagrina margaritifera*, apparently in its natural condition, which in places shows comparatively coarse parallel lines on its surface, but have examined numerous pearls without finding any such lines present, although under high powers of the microscope the surface is by no means smooth, but as soon as a small facet was ground on the pearl the typical wavy lines appeared and could be seen side by side with the unabraded surface in same field of view.

Furthermore, the brilliancy of a pearl section is increased by mounting in balsam, instead of being suppressed, as would be the case were it due to corrugations on the surface, and if a nearly diametrical section of a round pearl is observed by reflected light, the pearly luster will be confined to a small spot near the center, where the laminae are approximately parallel with the surface of the section. Brewster mentions that the distance between the grooves varies from a two hundredth to a five thousandth part of an inch, while Carpenter states that they may be as close as a seventy-five hundredth of an inch, both measurements evidently made on oblique sections, as I have carefully counted the laminae in a section of *Unio* pearl at right angles to the surface and found them to range between 54,000 and 57,000 to the inch. This brings the luster of pearls within the range of Lord Rayleigh's explanation of colors due to repeated laminae, which deduction may be confirmed by the micro-spectroscope.

In round pearls used as gems the patches of color are generally too small and intermingled to permit of satisfactory investigation in this manner, but in sections of pearl shell places can readily be found where the laminae are nearly parallel to the surface and sufficiently flat to exhibit an area of uniform color large enough to produce a good spectrum, and the single bands of color shown are sometimes

almost as sharp as those from opal, proving that the colors are due to the repeated parallel laminæ and not to the lines or corrugations, although the latter may be the only visible indications of structure when viewed with the microscope in the usual manner.

While they probably know or care little about optical science, the fabricators of false gems have not failed to note the resemblance between the colors of pearl shell and those of opal, and an imitation of opal used in cheap jewelry is made by cementing a thin section of pearl shell between two rounded and polished pieces of glass.